SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

LABORATORY SERVICES BRANCH

SCAQMD METHOD 317-93

QUALITATIVE ANALYSIS OF NATURAL OR MAN-MADE FIBER TYPES

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This method applies to the determination of natural fibers in samples as regulated and defined by Rule 1130.1.

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1.0 Principle

1.1 This method is intended to analyze a wide variety of fibers. All techniques to determine the properties cannot be documented in this method. Instead, several reference sources are suggested in this method that document the properties of fibers which may be observed and explain in detail the techniques necessary to determine the optical properties. For this reason, only analysts who have satisfied the criteria in Section 6.1 should conduct this method of analysis.

1.2 This method is for the qualitative identification of fibers to differentiate between natural and man-made fibers as defined by SCAQMD Rule 1130.1. The analysis is based on microscopic observation of morphology by low power stereo binocular microscopy (SBM) and optical properties by polarized light microscopy (PLM). Identification is made by comparison with reference material of known fiber type and/or reference of published optical properties of fiber types.

2.0 Equipment

2.1 Hood equipped with HEPA filter (required if sample contains asbestos fibers).

2.2 Low power stereo binocular microscope, 10X - 30X magnification.

2.3 Polarizing light microscope (PLM) with 360^{o} rotating stage equipped with transmitted and reflected light capabilities.

2.4 Dispersion staining objective, central stop, 10X.

2.5 Compensator plate, 530 nm retardation.

2.6 Objective lenses, 10X, 20X and 40X.

2.7 Forceps and dissecting needles.

2.8 Microscope slides, standard, and coverslips type no. 1.

2.9 Petri dishes.

2.10 Reference fiber standards, bulk or slides, of known fiber types which can be purchased through microscope and accessory supply companies.

3.0 Reagents

3.1 Refractive index liquids, 1.400 - 1.700, in increments of 0.004.

3.2 Solvents, reagents and acids needed to prepare samples (reagent grade or as specified in reference).

4.0 Analytical Procedure

4.1 Sample preparation

4.1.1 Sample must be dry before starting. Expose material to ambient conditions or use a low temperature oven or heat lamp, if necessary.

4.1.2 Make a preliminary scan of the sample using the SBM. If higher magnification is necessary, scan the sample using the PLM with the 10X objective. Observe and record properties such as presence of different types of fibers, color, texture, sheen, and presence of extraneous material such as dyes, resins and coatings.

4.1.3 Note the presence of fibers that do not appear to be an integral part of the sample. These fibers may appear to be loosely associated, easily picked off , and not interwoven within the sample. Regard these fibers as background and not as an integral part of the sample.

4.1.4 Nonfibrous extraneous materials that may interfere with fiber identification may be removed as outlined in *ASTM D 629*, Section 9.

4.1.5 Consider only fibers that are integral parts of the sample. Extract and separate visually different types of fibers from the sample using forceps. If mechanical separation is impractical or proves difficult, see Table 2 of *ASTM D 629* for a list of solubilities of fibers in solvents.

4.1.6 A rapid preliminary identification of fiber types may be made by referring to *ASTM F 71*.

4.2 Identification

4.2.1 Cut fibers to lengths of 5 cm or less. Mount in 1.55 refractive index liquid.

4.2.2 Examine the extracted fibers under the PLM and record the following optical properties. For detailed procedures on performing determining the optical properties, see *The Particle Atlas*, Edition 2.

a) Morphology: Record physical characteristics such as texture, size, color, pleochroism, transparency, presence of inclusions, and occurrence of bundles.

b) Refractive index: Measure the refractive index of the fiber by Becke test.

c) Sign of elongation: Use the 530 nm retardation compensator plate.

d) Birefringence: Calculate the birefringence by the numerical difference between the refractive indices along the fiber axis and perpendicular to the fiber axis. Alternatively, from the interference colors observed under cross-polars, the birefringence can be approximated by means of the Michel-Levy birefringence chart (see *The Particle Atlas*). The interference colors are also dependent on the orientation and thickness of the fiber and are used to qualitatively determine placement in one of the four categories listed below.

Qualitative	Quantitative (N-n)
none	0.00 or isotropic
low	0.010
moderate	0.010-0.050
high	>0.050

e) Extinction: Under cross polars, determine the extinction angle of the fiber along its axis. Observe if the fibers disappear when aligned parallel to or at an angle from the vibration axis of the polarizer.

f) Dispersion staining colors: Use a central stop dispersion staining objective and record the colors of the fiber at both parallel and perpendicular orientations.

4.2.3 Compare morphology and optical properties to Tables 1 and 2 and/or to standard reference materials for identification.

5.0 Documentation

5.1 In the analyst's notebook, document sample source, lab number, date sample received, date analysis started, and sample description.

5.2 Document morphology as described in section 4.1.2 and optical properties as described in section 4.2.1.

5.3 Record and report the type of fiber(s) present as an integral part of the sample.

6.0 Quality Assurance/Quality Control

6.1 Analysts using this method must have demonstrated training and proficiency in fiber identification by satisfying at least one of the following criteria.

6.1.1 Successful completion of two courses in the identification of particles and fibers by polarized light microscopy.

6.1.2 In-house training and one year experience in the identification of particles and fibers.

6.2 A second analyst must review the results and sign and date the notebook.

6.3 The laboratory must have available and within access of the analyst, reference manuals and reference materials necessary for fiber identification.

7.0 References

1) McCrone,W.C., Delly, J.G., *The Particle Atlas, Edition II.*

2) ASTM D 276-87, *Method for Identification of Fibers in Textiles.*

3) ASTM D 629-88, *Standard Test Methods for Quantitative Analysis of Textiles.*

4) ASTM 71-68, *Standard Practice for Using the Morphological Key for the Rapid Identification of Fibers for Contamination Control in Electronic Devices and Microelectronics*.

5) Perkins, R.L., Harvey, B.W.; *EPA Test Method: Method for the Determination of Asbestos in Bulk Building Materials*; EPA/600/R-93/116; July 1993.

TABLE 1 OPTICAL PROPERTIES OF NATURAL FIBERS*.

Type	Morphology	Refractive Index	Sign of Elongation	Bire-fringence	Extinction	C.S. Dispersion Staining Colors
Cotton, mercerized linters	Transparent, colorless, twisted. Lustrous, transparent, colorless, slightly twisted. Buff, transparent, fuzzy, short. May contain crook, hooks, wartlike growths.	length 1.578 cross 1.532 length 1.544-1.566 cross 1.522-1.524	+	0.046 0.030-0.044	none (due to fiber twist)	In 1.55 R.I. liquid ∥ pale yellow ⊥ pale blue
Flax	Colorless to pale yellow bast fibers, straight. Contains transverse nodes in shapes of I's, X's, V's and Y's.	length 1.594 cross 1.532	+	0.062	parallel	In 1.55 R.I. liquid ∥ pale yellow ⊥ pale blue
Hemp	Bast fibers, colorless with surface irregularities such as joints, fractures and swollen tissues.	length 1.585-1.591 cross 1.526-1.530	+	0.06	parallel	In 1.55 R.I. liquid ∥ pale yellow ⊥ pale blue
Ramie	Transparent, colorless, approx. cylindrical. Have nodelike ridges and longitudinal striations. No twist.	length 1.596 cross 1.528	+	0.068	parallel	In 1.55 R.I. liquid ∥ pale yellow ⊥ pale blue
Animal	Generally characterized by scales, presence of central canal, or medulla. Transparent.	length 1.55 cross 1.54	+	0.01	parallel	In 1.55 R.I. liquid ∥ magenta ⊥ blue
Silk (Tussah)	Pale yellow to yellow-brown, transparent, looks like broad continuous ribbons but are thin wedge shapes at cross section.	length 1.59 cross 1.54	+	0.05	parallel	In 1.55 R.I. liquid ∥ pale yellow ⊥ pale blue
Silk (cultivated)	Transparent, colorless. Appears smooth, continuous cylinders of constant diameters.	length 1.591 cross 1.538	+	0.053	parallel	In 1.55 R.I. liquid ∥ pale yellow ⊥ pale blue
Wool	Prominent, overlapping scales, transparent, colorless cylinders. Medulla may be present; usually absent.	length 1.556 cross 1.547	+	0.009	parallel	In 1.55 R.I. liquid ∥ magenta ⊥ blue

*Compiled from The Particle Atlas.

TABLE 2 OPTICAL PROPERTIES OF MAN-MADE FIBERS*.

Type	Morphology	Refractive Index	Sign of Elongation	Bire-fringence	Extinction	C.S. Dispersion Staining Colors
Acetate	Continuous striations with two to four smoothly rounded lobes. Contains no delustering pigments, hence, "bright".	length 1.478 cross 1.473	+	0.005	parallel	In 1.55 R.I. liquid || white ⊥ white
Rayon, viscose	Continuous, transparent, colorless, striated cylinders with 6-20 rounded lobes.	length 1.535-1.555 cross 1.515-1.535	+	0.020	parallel	In 1.55 R.I. liquid || blue ⊥ pale blue
cupramm-onium	Continuous, transparent, colorless with no surface markings. Shows numerous flattened sections.	length 1.548-1.553 cross 1.519-1.527	+	0.021-0.034		
Triacetate (*Arnel*)	Parallel striations, transparent, colorless, smooth, continuous cylinders. Four or more lobes.	length 1.469 cross 1.469	+	0.001	parallel	In 1.55 R.I. liquid || white ⊥ white
Acrylic (*Orlon*)	Transparent, smooth, joined cylinder pairs. Appears to have a central longitudinal striation.	length 1.505-1.515 cross 1.507-1.517	-	0.002	parallel	In 1.55 R.I. liquid || pale blue ⊥ pale blue
(*Acrilan*)	Similar to Orlon but round to off-round, single cylinder.	length 1.520 cross 1.525	-	0.005		
Aramid (*Kevlar*)	Pale yellow, transparent smooth cylinder. Characterized by wrinkled lines and spiral banding.	length 2.35 cross 1.641	+	0.71	parallel	In 1.605 R.I.liquid || white ⊥ yellow
Modacrylic (*Dynel*)	Continuous, transparent, colorless with a deeply fluted surface. Dog-bone, peanut, and three-lobe shapes in cross section.	length 1.535 cross 1.533	+	0.002	parallel	In 1.55 R.I. liquid || pale blue ⊥ pale blue
Nylon (*Caprolan*)	Smooth continuous transparent, colorless cylinders. May contain delustering agent.	length 1.580 cross 1.520	+	0.060	parallel	In 1.55 R.I. liquid || pale yellow ⊥ pale blue
(*Nomex*)	Transparent, colorless, continuous. High temperature resistance.	length 1.747->1.8 cross 1.664-1.680	+	0.036 or higher		|| white ⊥ yellow
(*Tynek*)	General appearance of mechanically shredded plastic ribbon. Frayed fibers are round or ribbonlike.	indices 1.52-1.56	+	0.06		
Olefin (*Courlene Py*)	Colorless, transparent, smooth walled cylinders.	length 1.530 cross 1.496	+	0.034	parallel	In 1.55 R.I. liquid || pale blue ⊥ pale blue
Polyester (*Dacron*)	Continuous, transparent, colorless cylinders. Smooth surface except where delustering agents have broken through.	length 1.710 cross 1.535	+	0.175	parallel	In 1.55 R.I. liquid || white ⊥ pale blue
Spandex (*Lycra*)	An elastomer having 500-600% elongation with nearly instantaneous recovery. Longitudinal striations due to multifilament.	length 1.56 cross 1.56	+	very low	parallel	
Glass, fiber glass	Transparent, colorless, continuous, almost always smooth and regular. Isotropic.	normally 1.55				In 1.55 R.I. liquid blue
Fluorocarbo n (*Teflon*)	Translucent, brown, continuous cylinders.	indices 1.34-1.38		low	parallel	In 1.55 R.I. liquid - white
Saran (*Saran*)	Transparent, colorless continuous. Uniformly round, smooth and unmarked surface.	indices 1.60-1.63	-	low	parallel	In 1.55

*Compiled from The Particle Atlas. Names in italics are trademarks.